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Caking behaviour of food powder binary mixes containing sticky and non-sticky powders

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Research Highlights

- At least 20% “sticky” powder fraction was required to initiate caking.
- Different “non-sticky” powders can display very different caking behaviours in mixes depending on the particular “sticky” powder.
- All “non-sticky” powders greatly reduced the strong caking effect of the “stickiest” maltodextrin powder.
- Caking behaviour is highly influenced by the “sticky” powders ability to deform and flow.

Caking behaviour of food powder binary mixes containing sticky and non-sticky powders

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ABSTRACT

Caking of food powders is highly undesirable. Many food powders are powder ingredient mixes and there is little work reported on the caking of food powder mixes. This study focusses on the caking of food powder binary mixes consisting of a “sticky” powder (whey permeate WP or maltodextrin MD) and a “non-sticky” powder (salt, flour or paprika). The powders were exposed to 76% relative humidity to make the WP and MD sticky. Force-displacement testing coupled with visual assessment of 2 particles in contact using a microscope were used to investigate the caking behaviour of the binary mixes. A “sticky” powder mass fraction of at least 20% was required to initiate caking and formation of weak cakes. Increasing percentage “sticky” powder fraction above the initial caking percentage resulted in progressively stronger cakes, however the rate of this progression was much less for the stickiest MD powder. The “non-sticky” powders and how they interacted with the “sticky” powders influenced the caking behaviour of the mix. For example, salt formed the strongest cakes in the WP mixes but formed the weakest in the MD mixes. Ability of a “sticky” powder to deform and flow influenced caking behaviour.

Keywords: Powders mixes; Powder caking; Glass transition.

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1. Introduction

Powder flow properties are important in handling and processing operations (Knowlton et al., 1994), as flow problems in hoppers and silos are commonplace problems for engineers and process operatives. Furthermore, bulk materials such as food powders can be very problematic whereby product may be rendered unusable due to severe caking. During handling, storage, processing and distribution to the final consumer, the powders may experience compaction and variations in temperatures and atmospheric humidities, which may alter the handling behaviour and appearance of the powders (Teunou and Fitzpatrick, 2000; Fitzpatrick et al., 2007). Water uptake by the powder is often a key cause of caking, thus it is very important to know the water sensitivity and the hygroscopicity of the powder components for predicting the kinetics of reduced flowability and caking (Fitzpatrick et al., 2010).

Many food powders contain amorphous glassy components, such as amorphous sugars and maltodextrins. The glass transition temperature (T_g) is very important because amorphous components may become sticky (Aguilera et al., 1995; Kudra, 2003; Adhikari et al., 2005; Descamps et al., 2013) if the powder temperature is elevated above its T_g , although the temperature above T_g does depend on the material, T_g definition (onset or midpoint) and test method used (Murti et al., 2006; Paterson et al., 2005; Foster et al., 2006). At these temperatures, powders develop viscous behaviour that may give rise to stickiness and caking. They may also flow into each other and form sinter bridges that give rise to very strong cakes (Palzer, 2005). Moisture content has a big influence on T_g with higher moistures leading to lower T_g . Consequently, caking of powder particles composed mainly of amorphous substances is strongly dependent on the relative

humidity of the surrounding air and temperature. Crystallisation may also occur if the powder temperature is greater than its T_g , especially for smaller molecules such as lactose whereby the molecules have sufficient mobility to initiate crystallisation (Jouppila and Roos, 1994; Jouppila et al., 1997; Roos, 2002; Lai and Schmidt, 1990). This will also impact on the caking behaviour and cake strength of a powder.

Food powder mixes can contain a variety of component powders and may cake when exposed to higher relative humidities or when compacted. The caking behaviour is often attributable to one or a small number of powders that become sticky and cause the mix to cake. Consequently, the susceptibility to caking will depend on the stickiness and fraction of “sticky” powder present and how it interacts with the other powders. There is a significant amount of work presented in the literature on the flowability and caking behaviour of individual powders. However, there is very little work reported on the caking of powder mixes. The focus on powder mixes is important, as many powder ingredients are formulated and supplied as a mix of different powders. The aim of this work is to investigate the caking behaviour of food powder mixes. In particular, how powder fractions and powder properties coupled with time and handling conditions influence the caking behaviours of powder mixes. This work focused on simple binary mixes consisting of a “sticky” powder and a “non-sticky” powder. The “sticky” powders were whey permeate and maltodextrin DE21; the “non-sticky” powders were salt, flour and paprika. The study investigated how the fraction of “sticky” powder, its stickiness and interaction with “non-sticky” powders influenced the caking behaviour of the binary powder mixes. A novel force-displacement tester was used to measure cake strength (Fitzpatrick et al., 2008). This was supplemented by the use of digital microscopy to

visually assess the adhesion of two powder particles in contact, similar to work reported by Feeney and Fitzpatrick (2011).

2. Materials and methods

2.1 Powders

A 25 kg bag of commercial spray-dried maltodextrin DE 21 (Glucidex® 21) was obtained from Roquette Frères, Lestrem, France. A 25 kg bag of commercial spray-dried whey permeate powder was obtained from Kerry Ingredients, Ireland. A wheat flour (Odlums) and sodium chloride salt (Saxo) was obtained from a local supermarket, and a paprika powder was obtained from a local spice powder supplier. Sodium chloride was obtained from Sigma-Aldrich Fluka for controlling relative humidity.

2.2 Powder properties

Moisture content of powders (% w/w) was measured using an infra-red moisture analyser (Sartorius MA 150). The median (d_{50}) of a volume particle size distribution was measured by laser diffraction using the Malvern Mastersizer MSS with powder feeder unit. Powder tapped bulk density was measured using an Engelsmann model A.-G. mechanical tapping device, where the volume of a given mass of powder was measured after 400 taps. The moisture content, particle size and bulk density of the powders tested are provided in Table 1.

The whey permeate and maltodextrin powders were chosen as “sticky” powders because they have amorphous components that when subjected to 76% relative humidity over time will sorb sufficient moisture to become sticky. The three “non-sticky” powders

(salt, flour and paprika) were chosen because they have different properties. The salt is a crystalline material, while the others are not. Table 1 shows that there is a major variation in moisture content with salt having a very low value of 0.06%, flour having a high value of over 10% and paprika having an in between value of nearly 6%. Table 1 also shows significant variations in particle size and bulk density.

2.3 Measurement of powder cake strength

An empirical tester for quantitatively measuring an index of cake strength was used (Fitzpatrick et al., 2008). It consisted of placing around 20 g of powder into a cylindrical aluminium dish which had a circular hole exactly in the centre of the dish with dimensions illustrated in Fig. 1a. A layer of aluminium foil was taped to the bottom of the dish to cover the hole, preventing powder falling through and to prevent exposure to atmosphere through the hole. The powder was spread out across the dish using a levelling device to give a thin powder layer of about 7 mm. Samples were then exposed to a relative humidity of 76% RH at 20°C over time to investigate how these conditions influence the cake strength of the powders. The relative humidity was achieved by placing a dish containing powder into a glass kilner jar (Fig. 1b), containing a saturated sodium chloride solution to obtain 76% RH, which was then sealed.

Following exposure, a dish was removed from the kilner jar, the foil was removed from beneath the aluminium dish and the dish was centred below a rod attached to a TA HD Plus Texture Analyser (Stable Microsystems, UK). The rod had a diameter of 5 mm and was initially moved downwards to just above the top of the caked powder. The rod was then moved downward through the caked powder at a constant speed of 30 mm/min

and the force versus displacement was measured using the TA Texture Analyser as the rod moved through the cake. As the rod engaged the surface of the cake, the force increased to a maximum and then gradually reduced as the rod continued to push a plug of caked powder through the hole in the centre of the dish. The peak force measured was defined as the cake strength. The water content of the caked sample was then measured by taking three samples of caked powder and measuring the water content.

2.4 Visual assessment of powder particle adhesion using microscopy

A light microscope capable of taking digital images (Bresser LCD Micro 40X-1600X) was used to take photographs of two powder particles in contact with each other when exposed to different relative humidities (Feeney and Fitzpatrick, 2011). Two powder particles were placed in contact with each other inside a transparent Petri dish. An open aluminium dish containing a saturated NaCl salt solution was also placed inside the Petri dish (to produce 76% relative humidity). The Petri dish was then sealed using a parafilm tape. Photographs were taken of the particles over time using the microscope to visually observe any changes in particle shape and formation of any bridges between the particles.

2.5 Experimental trials

The “sticky” powders were exposed to 76% RH over time to measure cake strength and moisture content over time. These data were used to investigate the caking behaviour and to identify an exposure time required to attain a high cake strength. These exposure times would then be used subsequently in the binary mixing trials with the “non-sticky” powders. The chosen exposure times for whey permeate and maltodextrin mixes were

chosen to be 8 hours and 6 days, respectively, and this is outlined in more detail in section 3. The “non-sticky” powders were also exposed to these time durations to ensure that the powders did not cake.

At the selected exposure times, binary powder mixes containing a “sticky” and “non-sticky” powder were formulated on a % “sticky” powder weight per total powder weight basis. These were formulated at compositions of 20%, 40%, 60%, 80% and 90% “sticky” powder. The mixing was done rapidly to prevent moisture sorption and each mix was placed in an aluminium dish and placed inside a kilner jar for the chosen exposure time, as described in section 2.3, after which cake strength and moisture content were measured. The tests were done in duplicate and the measured values are presented in the figures as error bars around the average values. Complementary visual assessment trials were conducted where photographs were taken over time of two particles in contact, as outlined in section 2.4.

3. Results and discussion

3.1 Caking behaviour of the sticky and non-sticky powders

The evolution of cake strength and moisture content over time for the “sticky” powders, whey permeate (WP) and maltodextrin (MD), is presented in Fig. 2. The WP attained a maximum cake strength of about 34 N after about 10 hour exposure and the moisture content increased from an initial moisture content of 1.9% to about 3.7%. After 10 hour exposure, cake strength decreased down to about 10 N after 24 hour exposure. The moisture content showed a decrease to about 3.2% from 10 to 14 hour exposure, but was then followed by an increase to around 5.1% after 24 hour exposure. The decrease is

indicative of some amorphous lactose crystallisation because crystalline lactose binds little water at the exposure conditions. The caking of WP is most likely due to stickiness associated with amorphous lactose absorbing moisture and its glass transition temperature (T_g) being reduced below the powder temperature of 20 °C. However, at the T_g of 20 °C, the equilibrium moisture content of amorphous lactose is about 7% (Roos, 2002). This is much greater than any of the moisture contents measured during the 24-hour test. The reason for this is possibly due to the WP powder containing a significant amount of lactose in the crystalline state, thus the amorphous lactose present is only a fraction of the lactose and the moisture content of the amorphous content is potentially much higher giving rise to stickiness.

Amorphous powder particles at temperatures above their T_g are no longer glassy solids and attain viscous properties which enable them to change shape and flow into each other forming sinter bridges between particles (Descamps et al., 2013; Palzer, 2005). Photographs were taken of two WP particles in contact over time when exposed to 76% RH, to observe if any bridging between particles or change in particle shape was taking place. This is illustrated in Figure 3a, which shows no significant change in particle shape or formation of sinter bridges. These results could be due to only a fraction of the lactose being in amorphous state giving rise to stickiness but the remaining lactose being in crystalline state along with other components present preventing flow and deformation of the WP particles.

The MD powder formed a much stronger cake of around 190 N and it took a lot longer time of about 5 to 6 days. There was a major increase in the moisture content from an initial moisture content of about 3.3% up to 17% after 7-day exposure. MD is a spray-

dried powder and is mainly in the amorphous form, thus its stickiness and strong cake formation is most likely due its glass transition temperature being reduced below the powder temperature (Descamps et al., 2013). At the T_g of 20 °C, the equilibrium moisture content of amorphous maltodextrin DE21 is about 10.7% (Descamps et al., 2013). At the measured high cake strengths on days 4 to day 7, the moisture content increased from 10 to 17%, which is within or above that at a T_g of 20 °C. Photographs were also taken of two MD particles in contact over time when exposed to 76% RH, to observe if any bridging between particles or change in particle shape was taking place. This is illustrated in Fig. 3a, where particle deformation and sinter bridging were observed and this gave rise to the very strong cake strengths measured.

As highlighted above, a major reason for conducting trials on the “sticky” powders alone was to identify an exposure time required to attain a high cake strength. From Figs. 2a and 2b, exposure times of 8 hours and 6 days were chosen for WP and MD, respectively. Repeatability tests were then carried out to ensure that there was no large variability in cake strength and moisture content at these times. Five replicates were conducted at these times and the mean and standard deviations are provided in Table 2, which show good repeatability at these exposure times.

The cake strengths and moisture contents of the “non-sticky” powders (salt, paprika and flour) were also measured when exposed to 76% RH over a 24-hour period and results are presented in Table 3. The powders did not cake over the 24-hour exposure time with no or very low strengths measured, the highest being 0.6 N for the paprika powder at 24-hour exposure (At strengths less than the 1 N, the powder is still a powder rather than a cake and has sufficient strength to span or form a cohesive bridge across the

hole in the bottom of the aluminium dish). This is to ensure that they remain non-sticky during testing. These powders also did not deform or change shape, as illustrated by the visual assessment in Fig. 3b.

3.2 Caking behaviour of binary mixes containing whey permeate powder

The effect of mixture composition on cake strength and moisture content, after 8 hour exposure to 76% RH at 20 °C, is presented in Fig. 4. The moisture content data did not display any unusual behaviour as the moisture contents of the mixes with higher whey permeate powder fraction moved progressively towards the moisture content of whey permeate powder, as illustrated in Fig. 4a. As expected, increasing the “sticky” whey permeate powder fraction does impact on the caking behaviour of the mixes, but it is not a linear relationship and it does depend on the “non-sticky” powder, as illustrated in Fig. 4b. These results show that at a WP powder fraction of 20%, there was no caking with measured cake strengths being very low at less than 1 N. At 40% WP fraction, weak cakes formed with cake strengths of about 2.5 N for both flour and paprika and 7 N for salt. The net result from these observations is that caking did not occur up to 20% fraction of the “sticky” WP powder and a weak cake started to form somewhere between 20 and 40% WP powder fraction.

Fig. 4b shows that the caking behaviour varies depending on the “non-sticky” powder in the mixes, with salt forming the strongest cakes and paprika forming the weakest. For example, at 80% WP powder fraction, the cake strengths of the mixes as a % of the cake strength of 100% WP were 72% (salt), 64% (flour) and 20% (paprika). Photographs are presented in Fig. 5 of a “sticky” and “non-sticky” powder particles in contact when

exposed to 76% RH. These show no noticeable changes in particle shape or contact area between particles, thus no high viscosity flow or bridging was observed. This is somewhat expected considering the photographs presented in Fig. 3. Consequently, the caking is most likely due to stickiness being developed by the amorphous lactose on the surface of the whey permeate powder particles. There are no obvious reasons for the variation in cake strength between the mixes. It might be related to the different moisture content of the mixes and the intrinsic surface chemistry of different powders giving rise to variations in attractive forces. It might be influenced by powder particle contacts within the powder bed, which in-turn is influenced by particle sizes, shapes and flowability.

3.3 Caking behaviour of binary mixes containing maltodextrin powder

The effect of increasing the “sticky” MD powder fraction on the caking behaviour of the mixes, after 6 day exposure to 76% RH at 20 °C, is presented in Fig. 6a. This is somewhat similar to the WP presented in Fig. 4b, showing a non-linear relationship. At 6-day exposure, the moisture contents of the “non-sticky” powders are all relatively high as presented in Table 3 and being about 14.3% for MD powder. For the mixes, the moisture contents behaved similarly to WP, as illustrated in Fig. 6b, with the moisture contents approaching that of MD at higher MD contents. The cake strength results show that at a MD content of 20%, there was no or little caking with measured cake strengths being less than 1 N for the salt (0.3N) and paprika (0.6 N), and being a little greater at 2N for the flour mix. This is somewhat similar to WP. At 40% MD fraction, cakes formed with cake strengths of about 10 N and 6 N for flour and paprika, respectively, however

the salt still did not cake, having a measured cake strength of 0.4 N which is less than 1 N.

The cake strengths of the MD/flour and MD/paprika mixes were greater than their WP counterpart mixes (Table 4), and this would be expected considering the large cake strength developed by 100% MD. However, the cake strengths of the mixes as a % of the 100% “sticky” powder were lower for the MD mixes, in particular for the salt and flour mixes. For example, the 80% MD fractions were only 8% for salt and 16% for paprika, which is much lower than their 80% WP mixes. For the WP mixes, the salt mixes formed the strongest cakes but formed the weakest cakes for the MD mixes. In fact, the MD/salt mixes produced weaker cakes than the WP/salt mixes.

Visual assessment of two particles in contact at 76% RH was undertaken, and the resulting photographs are presented in Fig. 7. Changes were expected because the MD particles did deform in Fig. 3. A major change occurred over time with the MD / salt particles. The MD deformed readily and flowed into the salt particle and eventually engulfed it, with the salt particle appearing to gradually dissolve within it. This liquid like behaviour may explain the weak cake strengths developed by the MD/salt mixes. It may also explain why the cake strengths of MD/salt mixes were much lower than the WP/salt mixes. For the MD / paprika particle contact, Fig. 7 shows the MD deforming over time and separating from the paprika particle. For the MD/flour, Fig. 7 shows 2 trials which show different phenomena. One MD/flour set of photos shows the MD deforming and increasing its association or contact with the flour, while the next set of MD/flour photos shows the MD deforming and detaching from the flour particle, much like the paprika. This suggests that when the MD deforms it may either coalesce more strongly with the

flour or paprika or separate from them, and this is possibly influenced by the initial shape, size and contact area of the particles. This behaviour may in-part explain the greatly reduced cake strengths of the flour and paprika mixes in comparison to the 100% MD powder, whereby some of the particle contacts may be lost in a powder bed due to separation caused by the deformation of MD. However, a major reason for the reduction in cake strength is most likely due to the reduction in the amount of strong sinter bridging between the MD particles themselves, due to there being less MD in a mix and consequently less MD/MD particle contact.

3.4 Caking behaviour of binary mixes containing MD and WP powders

A final set of trials were undertaken for binary mixes containing the two “sticky” powders (MD and WP). Mixes were formulated at 20%, 40% 60% and 80% MD powder fractions and exposed to 76% RH for 6 days. At 6-day exposure, the moisture content of WP and MD were about 12% and 14.3%, respectively. The moisture contents of the mixes were within this range. The cake strength of the MD/WP mixes is presented in Fig. 8a, along with those for the other MD mixes for comparison. The cake strength behaviour of the MD/WP mixes is somewhat similar to the other MD mixes. There was little or no caking at lower MD fractions up to 40% MD, followed by much stronger cakes at 60% and 80% MD. It might initially be surprising to have had little or no caking up to 40% MD considering that both powders are termed “sticky”, however it must be kept in mind that the exposure time was 6 days and the cake strength of WP significantly decreases after about 10 hours and is very low at about 5 N after 6 days. Thus, at 6-day exposure, the WP is behaving like a “non-sticky” powder, most likely because much of its

amorphous lactose has crystallised. Consequently, this may explain why MD/WP is behaving somewhat similarly to the other MD/"non-sticky" mixes at lower MD powder fractions.

At 60% and 80% MD, the MD/WP mix developed much stronger cake strengths than the other MD mixes, as illustrated in Fig. 8a. This may in-part be explained by the visual assessment of the MD and WP powder particles in contact presented in Fig. 8b. Here, the MD particle deforms over time, as expected, and the WP particle does not. Fig. 8b shows the MD particle flowing onto the WP particle over time, resulting in a large bridge between the 2 particles. The existence of this type of bridge may explain the higher cake strengths obtained at 60% and 80% MD in the MD/WP mixes as compared to the other MD mixes.

4. Conclusions

The application of a force-displacement test for measuring cake strength coupled with visual assessment of two particles in contact is a useful approach for investigating the caking behaviour of powders. The two "sticky" powders used in this study behaved somewhat differently, insofar as the WP became sticky but did not deform, while the MD did deform which enabled it form sinter bridges between MD particles. Consequently, the MD powder formed much a stronger cake than the WP powder.

In relation to the "sticky" / "non-sticky" powder mixes, a "sticky" powder mass fraction of at least 20% was required to initiate caking and formation of weak cakes. As expected, increasing percentage "sticky" powder fraction above the initial caking percentage resulted in progressively stronger cakes. For the MD mixes, the cake strengths

of the flour and paprika were stronger than their corresponding WP mixes, which was expected as the MD inherently forms stronger cakes. However, cake strengths developed as a percentage of 100% “sticky” powder were less for the MD mixes, thus the “non-sticky” powders had a greater impact on reducing the cake strength of the more “sticky” MD powder. This is most likely due to a reduction in the number of contacts between MD particles and thus a reduction in the amount of the strong MD / MD sinter bridges.

The particular “non-sticky” powder had an influence on the cake strength of the mix. For example, the salt powder displayed very different behaviour for the WP and MD mixes, forming the strongest cakes for WP mixes and the weakest for MD mixes. The visual assessment of two particles in contact contributed to trying to interpret these differences, as the caking behaviour is influenced by the “sticky” powders ability to deform and flow, which can be captured using a digital microscope.

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Fig. 1. (a) Aluminium dish used in force-displacement cake strength determination (with central hole and foil cover underneath to prevent powder falling through). (b) kilner jars used in the control of relative humidity using a saturated NaCl solution.

Fig. 2. Evolution of powder cake strength and moisture content over time at 76% RH (20 °C): (a) whey permeate; (b) maltodextrin.

Fig. 3. Photographs of 2 powder particles in contact over time when exposed to 76% RH (20 °C): (a) “sticky” powders; (b) “non-sticky” powders.

Fig. 4. Effect of whey permeate powder fraction on the caking of powder mixes (8 hour exposure to 76% RH at 20 °C).

Fig. 5. Photographs of whey permeate powder particle [particle at the bottom] in contact with a non sticky powder particle when exposed to 76% RH (20 °C) over time: (a) salt; (b) flour; (c) paprika.

Fig. 6. Effect of maltodextrin powder fraction on the caking of powder mixes (6 day exposure to 76% RH at 20 °C).

Fig. 7. Photographs of maltodextrin powder particle [particle on the left] in contact with a non sticky powder particle when exposed to 76% RH (20 °C) over time: (a) salt; (b) flour; (c) paprika.

Fig. 8. Maltodextrin / whey permeate powder mixes (a) cake strength; (b) visual assessment [maltodextrin particle is on the left].

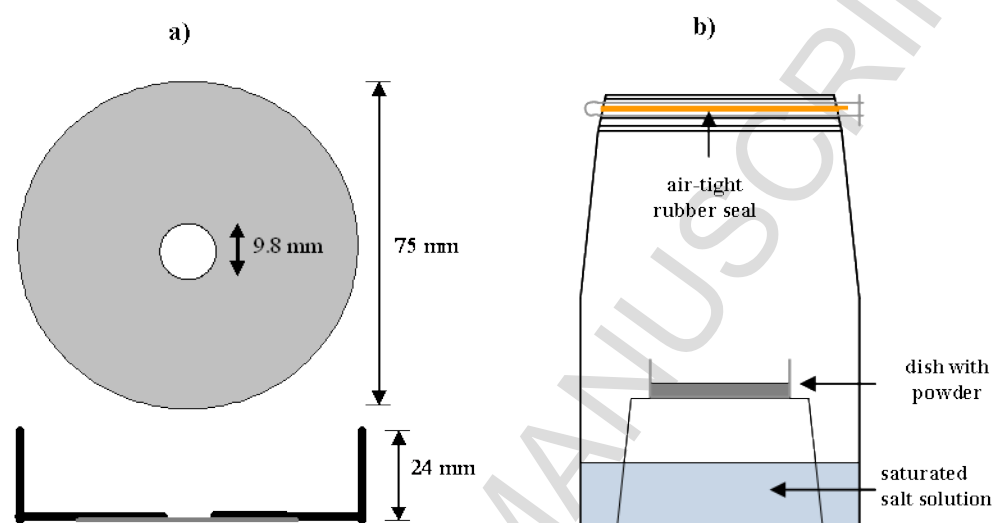
Fig. 1.

Fig. 2.

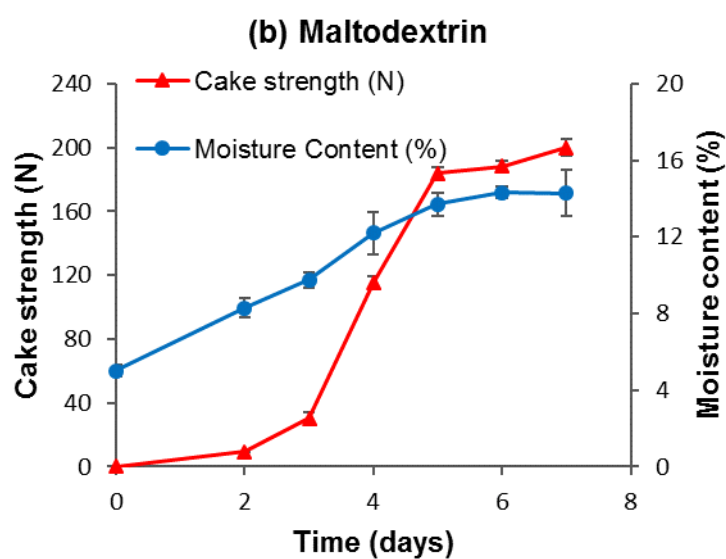
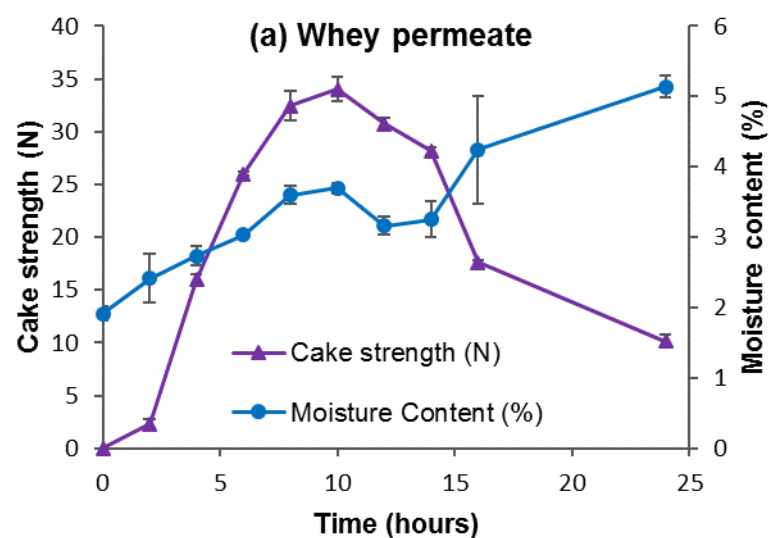


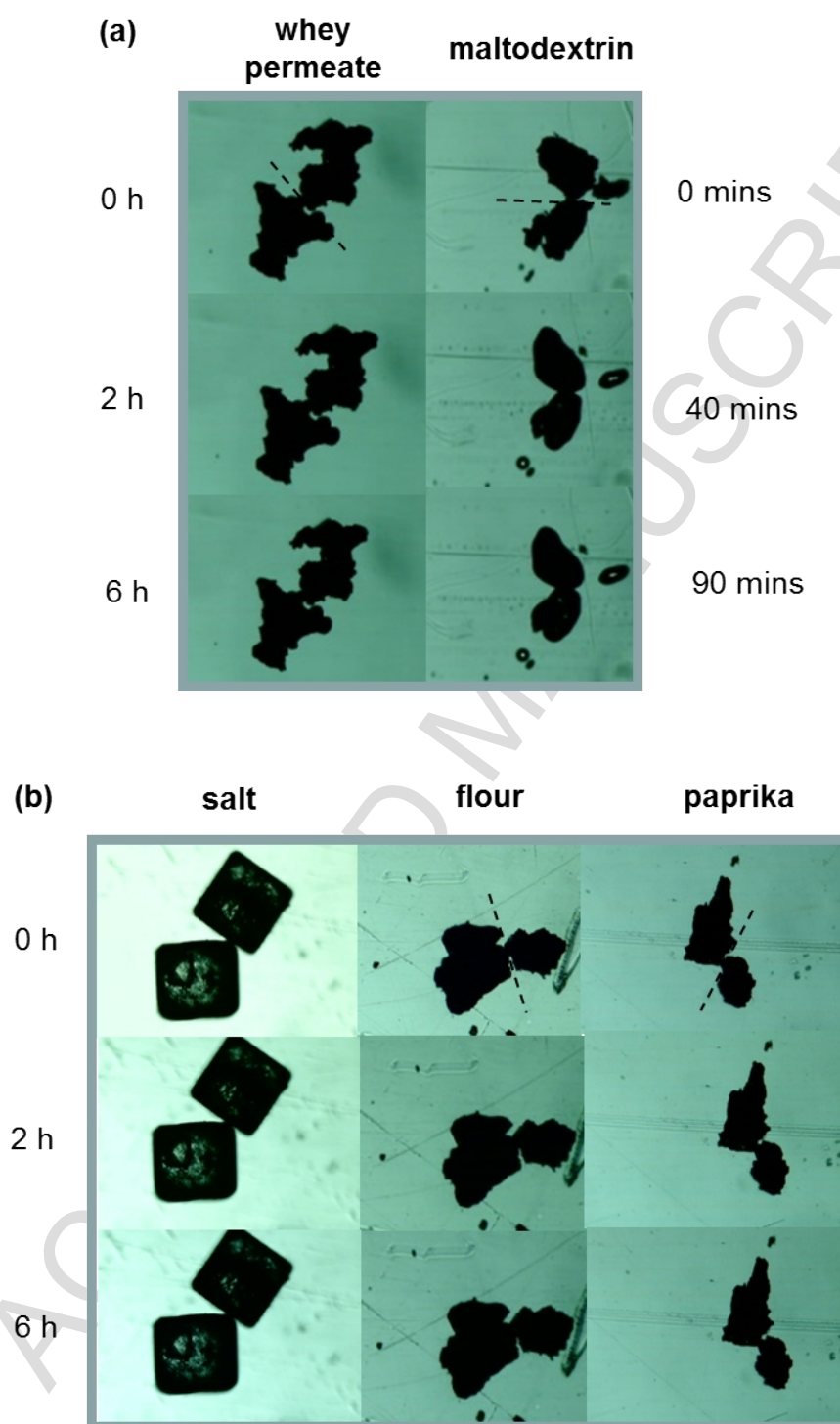
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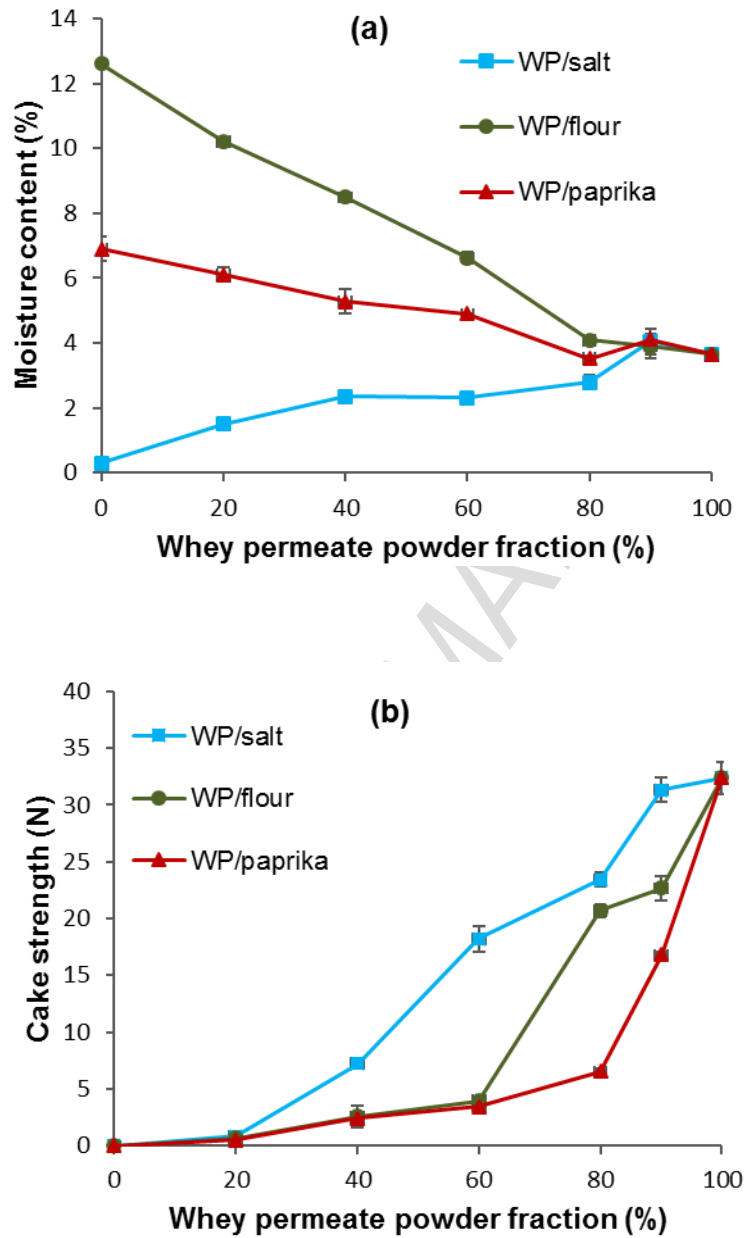
Fig. 4.

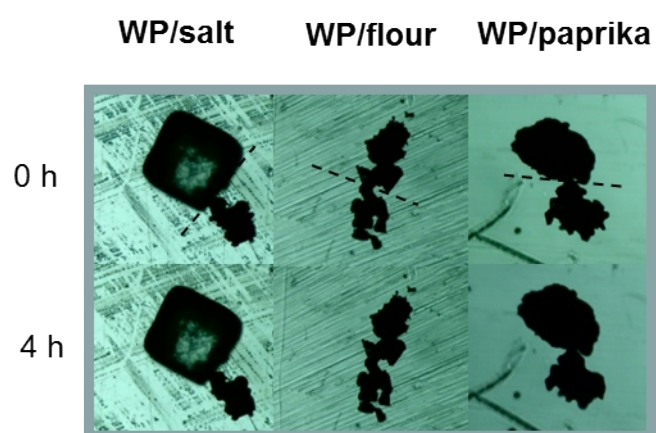
Fig. 5.

Fig. 6.

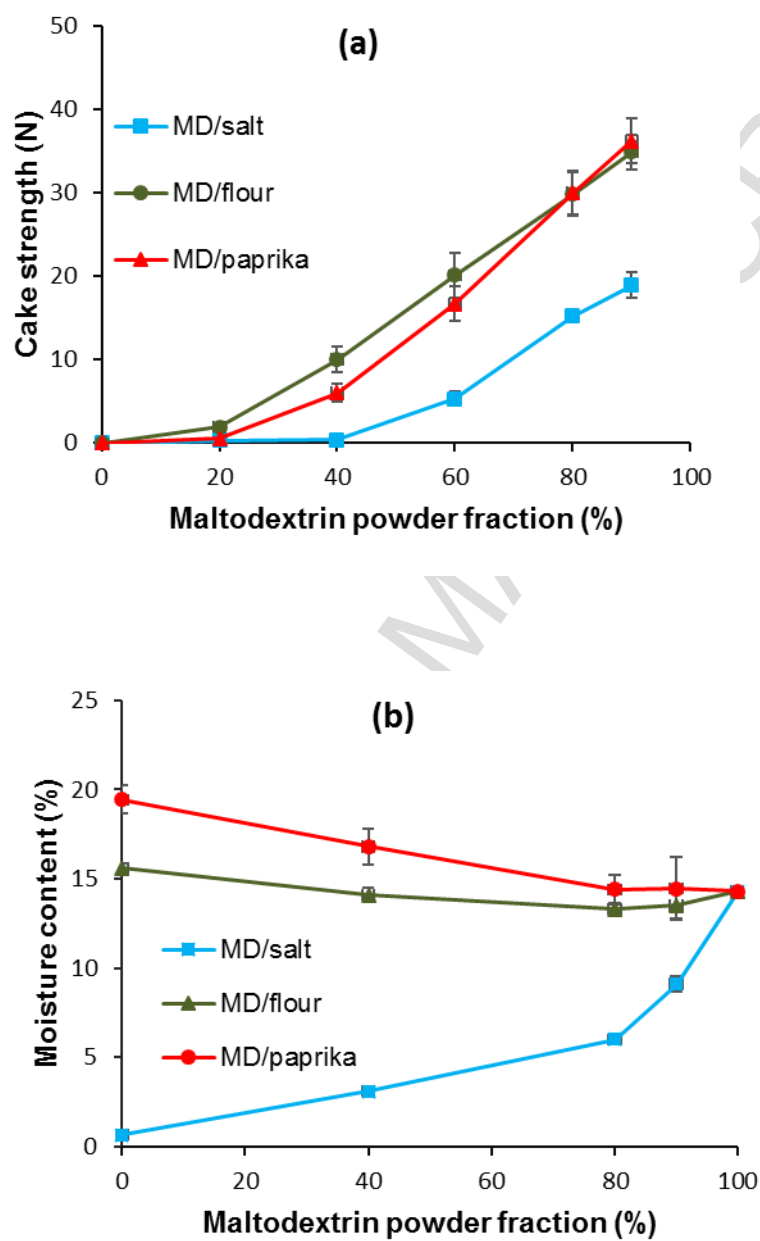


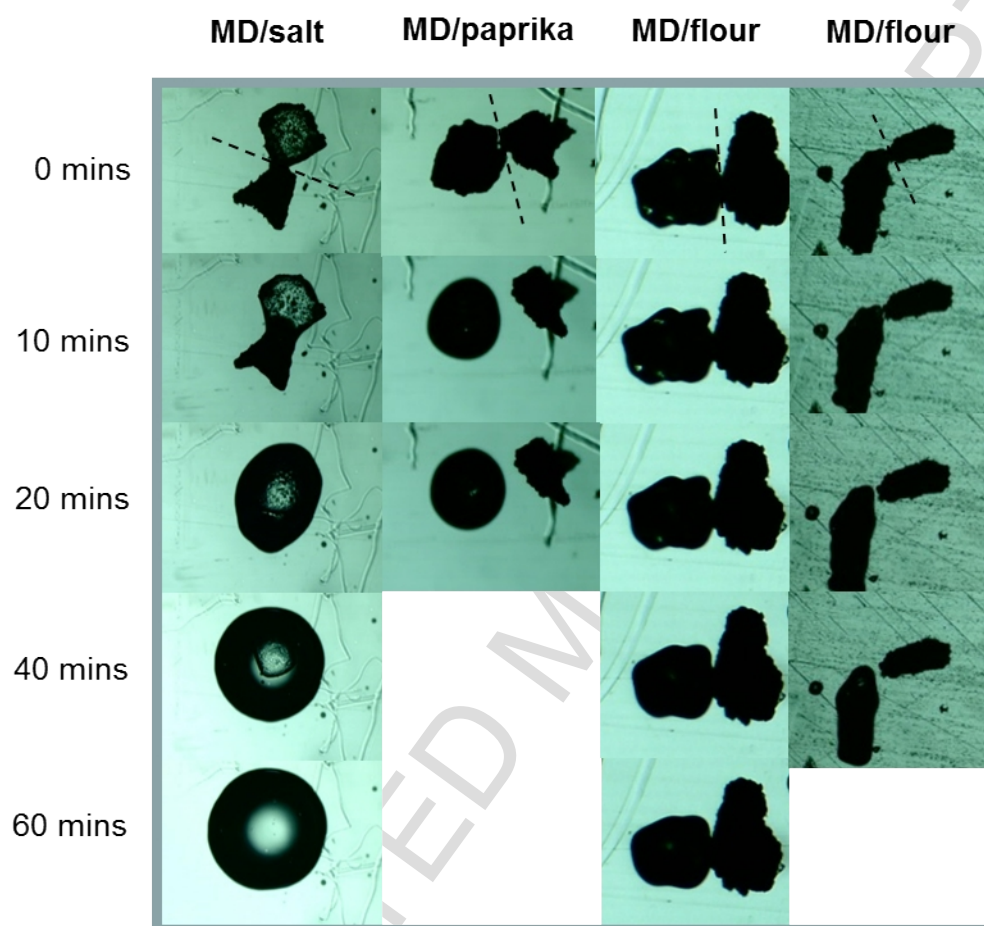
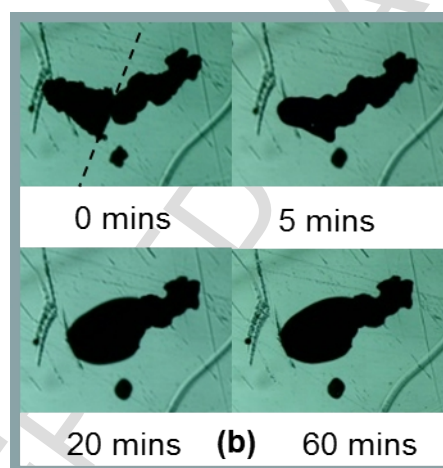
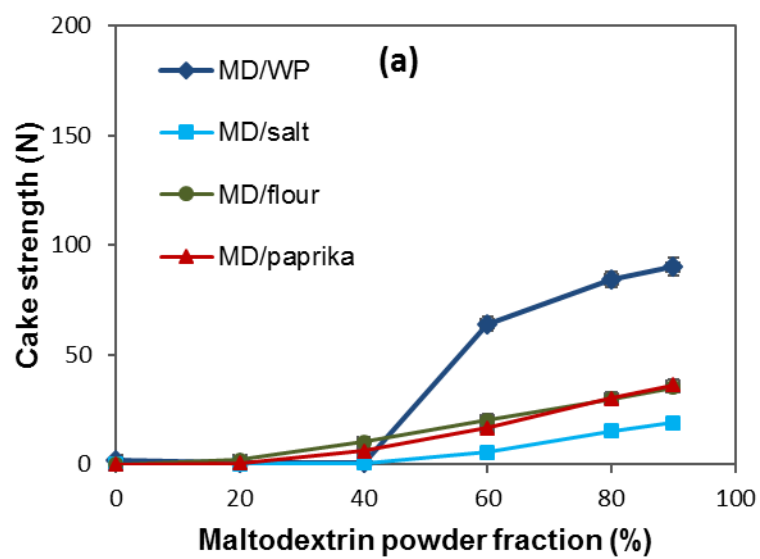
Fig. 7.

Fig. 8.



TABLES

Table 1. Powder moisture content, particle size and tapped bulk density

Powder	Moisture content (%)	Particle size d_{50} (microns)	Bulk density (kg L ⁻¹)
Whey permeate	1.9	92	0.93
Maltodextrin	3.3	110	0.31
Flour	10.6	65	0.77
Paprika	5.8	190	0.53
Salt	0.06	280	1.44

Table 2. Repeatability assessment of cake strength and moisture contents measurements [5 replicates].

	Whey permeate		Maltodextrin	
	Mean	Standard deviation	Mean	Standard deviation
Cake strength (N)	32.4	1.4	188	3.7
Moisture content (%)	3.65	0.13	14.3	0.31

Table 3. Cake strength and moisture content of “non-sticky” powders when exposed to 76% relative humidity (20 °C) over time.

	salt	flour	paprika
<u>Cake strength (N)</u>			
<u>Time</u>			
0 h	0	0	0
4 h	0	0	0.1
8 h	0	0	0.2
24 h	0.07	0.06	0.6
6 day	0.14	0.17	1.1
<u>Moisture content (%)</u>			
<u>Time</u>			
0 h	0.06	10.6	5.8
4 h	0.15	11.7	6.2
8 h	0.29	12.6	6.9
24 h	0.61	12.7	9.3
6 day	9.1	12.8	13.8

Table 4. Cake strengths (N) of whey permeate (WP) and maltodextrin (MD) powder mixes when exposed to 76% relative humidity (20 °C) [exposure time: 8 h for WP; 6 days for MD].

% “sticky” powder	<u>Salt mixes</u>		<u>Flour mixes</u>		<u>Paprika mixes</u>	
	WP	MD	WP	MD	WP	MD
0	0	0	0	0	0	0
20	0.8	0.3	0.7	1.9	0.5	0.6
40	7.2	0.4	2.6	10.0	2.4	6.0
60	18.2	5.3	4.0	20.1	3.4	16.7
80	23.4	15.2	20.7	29.8	6.5	30.0
90	31.3	18.9	22.7	34.9	16.8	36.2
100	32.4	188	32.4	188	32.4	188